# At-Line Recertification of Recycled TCE

**Nuclear Materials Technology Division**

For the near-term, trichloroethylene (TCE) will be utilized for fissile weapon component cleaning for War Reserve (WR) Pit Manufacturing. To minimize waste we are preparing to recycle TCE. Recycled TCE will require recertification before it can be used for parts cleaning. Analyses for triethylamine (TEA) stabilizer (1 to 200 ppm), water (<100 ppm), residue upon evaporation (<0.001%), and visualization (colorless and no particles) are required. We have been developing analytical methods for TCE that minimize waste and are glove box compatible.



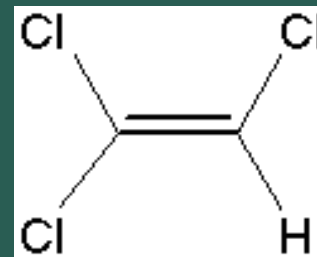FTIR system showing goal V-spheres and computer controls.



Structure of TCE

A small, dedicated, commercially available FTIR (SOC 400 FTIR) can be used to perform all these analyses. The unit will be mounted externally to the glove box and the IR signal will be transmitted through a sealed junction in the glove box wall through a light pipe to the sample.

The SOC 400 FTIR in combination with the V-sphere™ assembly can perform routine non-volatile residue analysis on TCE samples containing low concentrations of non-volatile hydrocarbon products. There was excellent correlation and linearity displayed over two orders of magnitude of oil residue as evidenced by a correlation coefficient of 0.999 for samples integrated between 2600 and 3200 $cm^{-1}$. The data collected indicated approximately 5 percent uncertainty at the 2.5-microgram level, well below the 10 ppm residue target maximum. This same data set revealed excellent sample stability over a 6-day period and represents a 95% reduction in sample usage.

A procedure was developed that determines TEA and water concentration in TCE using liquid-transmission FTIR in 5 or 10 mm quartz cells with peak area analysis between 2950 and 2987 $cm^{-1}$ for TEA and between 3626 and 3572 $cm^{-1}$ for water. The linear range of the method was 50 ppm to 1000 ppm TEA. Relative standard deviations of two precision studies with TEA were less than 3% and the method accuracy, as measured by percent difference, was within ± 5%. The detection limit for TEA was shown to be less than 1 ppm. The presence of hydrocarbon in the sample had a positive bias on the determination of TEA, however, this bias was < 10% at about 100 ppm hydrocarbons. The linear range of the method for water was from 25 to 230 ppm water or to about water saturated TCE. Method accuracy, as measured by percent difference, was within ± 10%. Since water needs to be confirmed to less than 100 ppm it was not necessary determine a minimum detection level. TEA and oil do not interfere with the analysis.

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